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(71) Applicant *(for all designated States except US)*: **PHILIP MORRIS PRODUCTS S.A.** [CH/CH]; Quai Jeanrenaud 3, CH-2000 Neuchatel (CH).

(72) Inventors; and

(75) Inventors/Applicants *(for US only)*: **WINTERSON, Warren, D.** [US/US]; 826 Lee Street, West Point, VA 23181 (US). **CUNNINGHAM, John, F.** [US/US]; 11811 Bollingbrook Drive, Richmond, VA 23236 (US).

(74) Agents: **SKIFF, Peter, K.** et al.; Burns, Doane, Swecker & Mathis, L.L.P., P.O. Box 1404, Alexandria, VA 22313-1404 (US).

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Declarations under Rule 4.17:

— *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii)) for the following designations AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZM, ZW, ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG)*

— *as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii)) for all designations*

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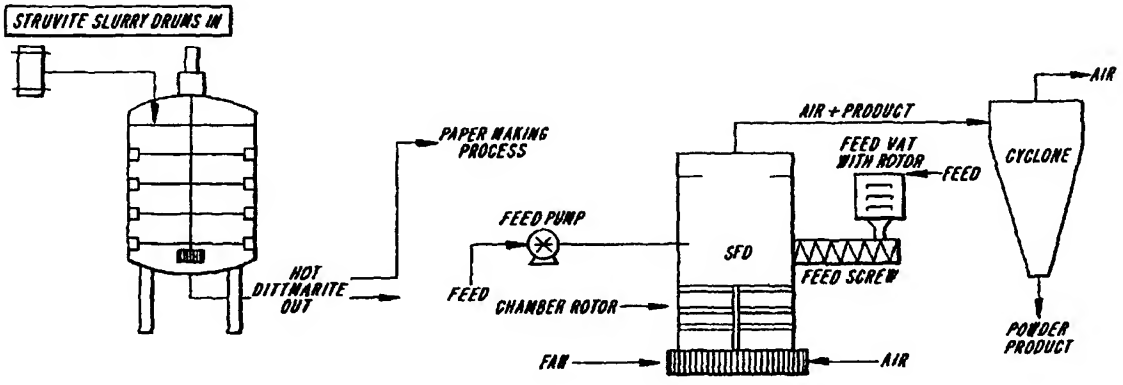
[Continued on next page]

(54) Title: PROCESS OF PRODUCING MAGNESIUM AMMONIUM PHOSPHATE IN MONOHYDRATE FORM (DITTMARITE)



(57) Abstract: A process for converting the more easily synthesized and stored AMP hexahydrate into monohydrate of high purity. The resultant monohydrate (dittmarite) can then be either dried to stabilize it, or used directly in cigarette production such as paper making as filler or a filler component together with calcium carbonate.

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— *before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

PROCESS OF PRODUCING MAGNESIUM AMMONIUM PHOSPHATE IN MONOHYDRATE FORM (DITTMARITE)

Field of the Invention

The present invention relates to methods of producing magnesium ammonium phosphate in monohydrate form (dittmarite), and in particular to methods of converting a hexahydrate form of magnesium ammonium phosphate (struvite) into a monohydrate form of magnesium ammonium phosphate monohydrate (dittmarite).

Background of the Invention

Ammonium magnesium phosphate (AMP) monohydrate has been discovered as useful in producing reduced harm smoking articles. Advantageous uses of AMP in smoking articles is described in commonly assigned U.S. Patent Application No. 09/399,159, which is incorporated herein by reference in its entirety.

Summary of the Invention

The present invention provides a process for converting the more easily synthesized and stored AMP hexahydrate into monohydrate of high purity. The resultant monohydrate (dittmarite) can then be either dried to stabilize it, or used directly in cigarette production such as paper making as filler or a filler component together with calcium carbonate.

More particularly, a preferred embodiment of the present invention provides a process wherein AMP hexahydrate is heated under pressure to convert it to the monohydrate form. In a preferred process, an aqueous slurry of AMP hexahydrate is heated in a pressure vessel (e.g., autoclave) to 100°C to 110°C with agitation under pressures from 1 psig to 15 psig.

Detailed Description of the Preferred Embodiments

Table 1 sets forth parameters used in preparing high purity AMP monohydrate (dittmarite) by converting AMP hexahydrate (struvite) under elevated temperature and pressure. The test runs were carried out using an aqueous slurry having a solids content of 35 wt. % struvite in autoclave equipment available from Lee Industries in Philipsburg, PA.

TABLE 1

Run	Temperature °C	Time Minutes	Pressure Psig	Volume Gallons
1	100	60	8	25
2	105	35 to 200	12	50
3	105	40	15	45

Runs 2 and 3 exhibited higher purity (i.e., higher conversion rates) results than Run 1, indicating the reaction should preferably be run at 105°C and 12 to 15 psig. The product of the reaction can be used as a slurry or the slurry can be dried to provide the dittmarite in particle form. In a paper making operation, the dittmarite can be supplied directly to a tank in which the dittmarite is mixed with one or more components of the paper making feedstock, preferably at a temperature and/or pressure sufficient to maintain stability of the reaction product. In a cigarette paper making process, the overall process steps can include:

Premix drums of struvite slurry

↓

Load drums of slurry into conversion vessel

↓

Run conversion process

↓

Pump out of conversion vessel into hot surge tank

↓

Pump dittmarite solution to paper making process

The pressure vessel has a jacket for heating and cooling, is rated for pressure operation, is of sanitary construction for easy clean up, and has at least one agitator, e.g. two agitators. One agitator scrapes the walls and creates a uniform temperature mixture, the second uses high shear to break up agglomerates and control particle size. This vessel is a preferred embodiment of the invention due to its high degree of temperature uniformity, and ability to control particle size, a critical parameter of paper making. Details of operating such a vessel can include:

Load drums of slurry into conversion vessel

↓

Start steam flow to vessel jacket to heat vessel and start vessel agitators

↓

Heat the mixture to the target temperature

↓

Hold at target temperature

↓

Pump resultant product to paper making machine or to spin flash drier

The conversion process is preferably run without intentional venting of pressure, or unintentional leaks, as undesirable side reactions may occur and the purity of the resultant product is reduced. Preferably the tank is in a filled condition during the conversion process. The conversion process is effective to produce at least 95%, preferably at least 95% or 98% or higher conversion of struvite to dittmarite. It is recognized that the initial slurry may contain additional ingredients and/or impurities that may be present provided such ingredients/impurities do not adversely affect the conversion process.

In a preferred process, a slurry of up to 50%, preferably 20 to 40 weight % solids of hexahydrate form of magnesium ammonium phosphate is heated under pressure of up to 25 psig, preferably 10 to 15 psig, for time sufficient to convert at

least 90%, preferably at least 95%, of the hexahydrate form of the magnesium ammonium phosphate to the monohydrate form thereof. The slurry can be heated to 90 to 135°C, preferably 100 to 110°C, for at least 5 minutes, preferably 5 to 25 minutes, to effect the conversion to monohydrate magnesium ammonium phosphate. The slurry can optionally be cooled but preferably not below a temperature at which the monohydrate magnesium ammonium phosphate will convert back to hexahydrate magnesium ammonium phosphate. Thus, the slurry of monohydrate magnesium ammonium phosphate is preferably maintained above 55°C, preferably above 60°C until the slurry is further processed such as by incorporation into paper or by drying the slurry to obtain particles of magnesium ammonium phosphate. FIG. 1 shows a layout of processing equipment suitable for effecting the conversion process and subsequent use of the dittmarite.

The monohydrate form of magnesium ammonium phosphate is desirable in paper making manufacture such as cigarette paper. The slurry of the monohydrate form of magnesium ammonium phosphate can be mixed with feedstock of a paper making machine or the slurry can be dried to particle form (e.g., powder) and such powder can be incorporated in the paper making feedstock. In order to prevent the magnesium ammonium phosphate in the monohydrate form from transforming back to the hexahydrate form, it is desirable to maintain the slurry above 55°C until it is incorporated directly in feedstock (preferably heated above 60°C) of the paper making machine or until the slurry is dried into particle form such as by flash drying which removes the water from the slurry under elevated temperature conditions. Once dry, the monohydrate form of the magnesium ammonium phosphate remains stable.

The struvite slurry hexahydrate magnesium ammonium phosphate can be obtained by any known processes of reacting magnesium hydroxide, ammonia, phosphoric acid and water. The present invention provides an advantageous and effective method of converting such slurry of hexahydrate form to the monohydrate form of magnesium ammonium phosphate usable to form filler for paper making

wherein a particle size is preferably in the range of approximately 2μm to 8μm, more preferably in the range of 2μm to 4μm. If the slurry is dried to particle form, a preferred spin-flash drier can be obtained from APV Anhydro of Tonawanda, New York.

While the invention has been described in detail with reference to specific embodiments thereof, it will be apparent to those skilled in the art that various changes and modifications can be made, and equivalents employed, without departing from the scope of the appended claims.

What is claimed is:

1. A process of making a monohydrate form of magnesium ammonium phosphate comprising:

heating a slurry comprising a hexahydrate form of magnesium ammonium phosphate under pressure for a time sufficient to convert the hexahydrate magnesium ammonium phosphate to the monohydrate form thereof.

2. The process as claimed in Claim 1, further comprising agitation of the slurry.

3. The process as claimed in Claims 1, wherein the slurry is heated to a temperature up to 150°C under a pressure of up to 100 psig.

4. The process as claimed in Claims 1, wherein the slurry is heated to a temperature of 100°C to 110°C under a pressure of about 10 to 15 psig.

5. The process as claimed in Claims 1, further comprising drying the slurry.

6. The process as claimed in Claim 5, wherein the drying step is conducted with a spin-flash drier.

7. The process as claimed in Claim 1, wherein the heating is carried out for a time sufficient to convert at least 95% of the magnesium ammonium phosphate in hexahydrate form to the monohydrate form thereof.

8. The process as claimed in Claim 1, wherein subsequent to the heating step the process further comprises cooling the slurry to a temperature above 55°C.

9. The process as claimed in Claim 1, wherein the slurry includes 20 to 40 weight % of solids content prior to the heating step.

10. The process as claimed in Claim 1, wherein the slurry is contained in a sealed vessel during the heating step.

11. The process as claimed in Claim 10, wherein the slurry is subjected to stirring during the heating step.

12. The process as claimed in Claim 1, wherein subsequent to the heating step the slurry containing the monohydrate form of the magnesium ammonium phosphate is mixed with paper making feedstock during a paper making process.

13. The process as claimed in Claim 12, the paper making process is a cigarette making process.

14. The process as claimed in Claim 1, wherein the heating is carried out for at least 5 minutes.

15. The process as claimed in Claim 1, wherein the heating is carried out for at least 10 minutes.

16. The process as claimed in Claim 1, wherein the heating is carried out for at least 15 minutes.

17. The process as claimed in Claim 1, wherein the slurry prior to the heating step has an average particle size of 4 to 7μm and/or the slurry has a pH of at least 7.

18. The product produced according to the process of Claim 1.

19. The product produced according to the process of Claim 5.

20. The product produced according to the process of Claim 13.

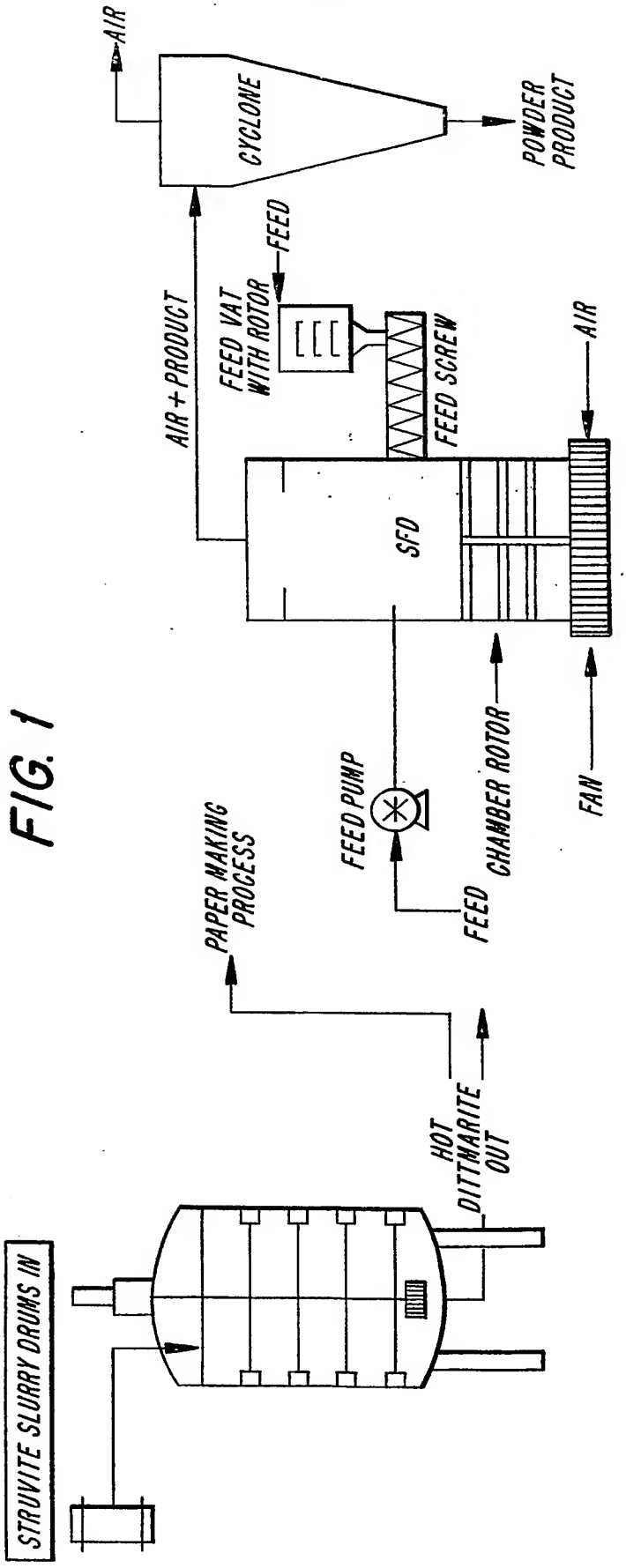

FIG. 1

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US02/24012

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) :C01B 25/45
US CL :423/306

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 423/306

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

NONE

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
T	US 2002/0114753 A1 (GREEN et al) 22 August 2002 (22-08-02), see [0008].	1-20
A	US 3,285,731 A (SALUTSKY et al) 15 November 1996 (15-11-66), see col. 3, line 18 to col. 5, line 60.	1-20
A	US 5,374,294 A (MOORE) 20 December 1994 (20-12-94), see col. 1, line 52 to col. 2, line 16.	1-20
A	US 5,294,348 A (HORNY et al) 15 March 1994 (15-03-94), see col. 1, line 56 to col. 2, line 65.	1-20
A -- X	US 3,348,910 A (GOODENOUGH et al) 24 October 1967 (24-10-67), see col. 1, line 12 to col. 3, line 3.	1-17 --- 18-20

[X] Further documents are listed in the continuation of Box C. [ ] See patent family annex.

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"A"	document defining the general state of the art which is not considered to be of particular relevance	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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"O"	document referring to an oral disclosure, use, exhibition or other means		
"P"	document published prior to the international filing date but later than the priority date claimed		

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4,777,026 A (GRIFFITH) 11 October 1988 (11-10-88), see col. 1, line 9 to col. 4, line 12.	1-20

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